

**Pacific Northwest  
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**Year 3 Post-Remediation  
Biomonitoring of Pesticides and  
Other Contaminants in Marine  
Waters Near the United  
Heckathorn Superfund Site,  
Richmond, California**

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Region 9  
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Pacific Northwest National Laboratory  
Richland, Washington

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YEAR 3 POST-REMEDIATION BIOMONITORING  
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IN MARINE WATERS NEAR THE UNITED  
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## SUMMARY

Marine sediment remediation at the United Heckathorn Superfund Site in Richmond, California was completed in April 1997. During February 2000, in Year 3 of post-remediation monitoring of marine areas near the United Heckathorn Site, water and mussel tissues were collected from four stations in and near Lauritzen Channel. Dieldrin and dichlorodiphenyl trichloroethane (DDT) were analyzed in water samples and in tissue samples from resident (i.e., naturally-occurring) mussels. In contrast to previous years, no mussels were transplanted to the study area in Year 3. Year 3 concentrations of dieldrin and total DDT in water and total DDT in tissue were compared with those from Years 1 and 2 of post-remediation monitoring (Antrim and Kohn 2000a,b<sup>1</sup>), and with preremediation data from the California State Mussel Watch Program (Rasmussen 1995) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (Lee et al. 1994). Year 3 water samples and mussel tissues were also analyzed for polychlorinated biphenyls (PCB), which were detected in sediment samples during Year 2 monitoring.

Mean chlorinated pesticide concentrations in some Year 3 water samples were higher than Year 2 levels and did not meet remediation goals. Mean total DDT concentrations in Year 3 water samples ranged from 1.9 ng/L to 5152 ng/L and exceeded Year 2 values at both Lauritzen Channel stations (Stations 303.2 and 303.3) and the remediation goal (0.59 ng/L) at all stations. Mean dieldrin concentrations in Year 3 water samples ranged from 1.45 ng/L to 1710 ng/L and were higher than the Year 2 values and the remediation goal (0.14 ng/L) at all stations. The highest concentrations of total DDT and dieldrin pesticides were found at Lauritzen Channel/End (Station 303.3). Detected PCB Aroclor 1254 concentrations ranged from 18 ng/L to 449 ng/L. The highest concentrations of dieldrin, total DDT, and Aroclor 1254 all occurred in a single sample (replicate b) collected from Lauritzen Channel/End. Excluding that particular replicate, the highest concentrations detected were 100 ng/L for dieldrin, 84.8 ng/L for total DDT, and 45.5 ng/L for Aroclor 1254.

Tissue analyses indicated that the bioavailability of chlorinated pesticides was generally similar in Year 3 to preremediation levels in the study area. Total DDT concentrations in mussel tissues measured in Year 3 were lower than preremediation levels at Lauritzen Channel/End and Santa Fe Channel/End (Station 303.4), but were higher than preremediation levels at Richmond Inner Harbor

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<sup>1</sup> Reports for Years 1 and 2 of post-remediation monitoring were revised and republished in July, 2000, after discovery of a reporting unit error in the original documents published in 1998 and 1999. Revised documents were distributed to all names on the original distribution list; they are also available on the web by searching for "Heckathorn" at <http://www.pnl.gov/main/publications>.

Channel (Station 303.1). Dieldrin concentrations measured in Year 3 were generally similar to prerediation levels at those stations for which prerediation levels were determined. The lowest mean total DDT and dieldrin levels measured in Year 3 were in tissues from Richmond Inner Harbor Channel (52  $\mu\text{g/kg}$  and 5.4  $\mu\text{g/kg}$  wet weight, respectively). Aroclor 1254 concentration was lowest at Santa Fe Channel/End (123  $\mu\text{g/kg}$  wet weight). Mean chlorinated pesticide concentrations were highest at Lauritzen Channel/End (522  $\mu\text{g/kg}$  total DDT and 42.7  $\mu\text{g/kg}$  dieldrin, wet weight). Aroclor 1254 concentration was highest at Lauritzen Channel /Mouth (Station 303.2; 187  $\mu\text{g/kg}$ , wet weight).

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## 1.0 INTRODUCTION

The United Heckathorn Site is located in Richmond Harbor, on the east side of San Francisco Bay in Contra Costa County, California (Figure 1.1). The site is an active marine shipping terminal operated by the Levin Richmond Terminal Corporation. The U.S. Environmental Protection Agency (EPA) listed the site on its National Priorities List of Federal Superfund sites because of chemical contamination of upland and marine sediments and because the site had the highest levels of dichlorodiphenyl trichloroethane (DDT) contamination measured during the California State Mussel Watch program (Rasmussen 1995). A remediation investigation of adjacent marine areas revealed widespread contamination of sediment by pesticides, particularly DDT and dieldrin (White et al. 1994). Significant pesticide contamination was limited to the soft, geologically recent deposits known as "younger bay mud." Pesticide concentrations were highest in Lauritzen Channel, and decreased with increasing distance from the former United Heckathorn Site, clearly indicating that Heckathorn was the source of contamination. An ecological risk assessment at the Heckathorn Site (Lee et al. 1994) reported data collected in 1991 and 1992 for contaminant concentrations in marine water, organisms, and sediments. This assessment revealed that DDT and dieldrin contamination originating from the United Heckathorn Site had been actively transported to offsite areas via surface waters.

Major components of the final remediation actions at the Heckathorn Site outlined in the Record of Decision (ROD 1996) are:

- dredging of all younger bay mud from Lauritzen Channel and Parr Canal, with offsite disposal of the dredged material
- placement of clean sand after dredging
- construction of a cap around the former Heckathorn facility to prevent erosion
- enactment of a deed restriction limiting use of the property at the former Heckathorn facility location to nonresidential uses
- marine monitoring to verify the effectiveness of the remediation.

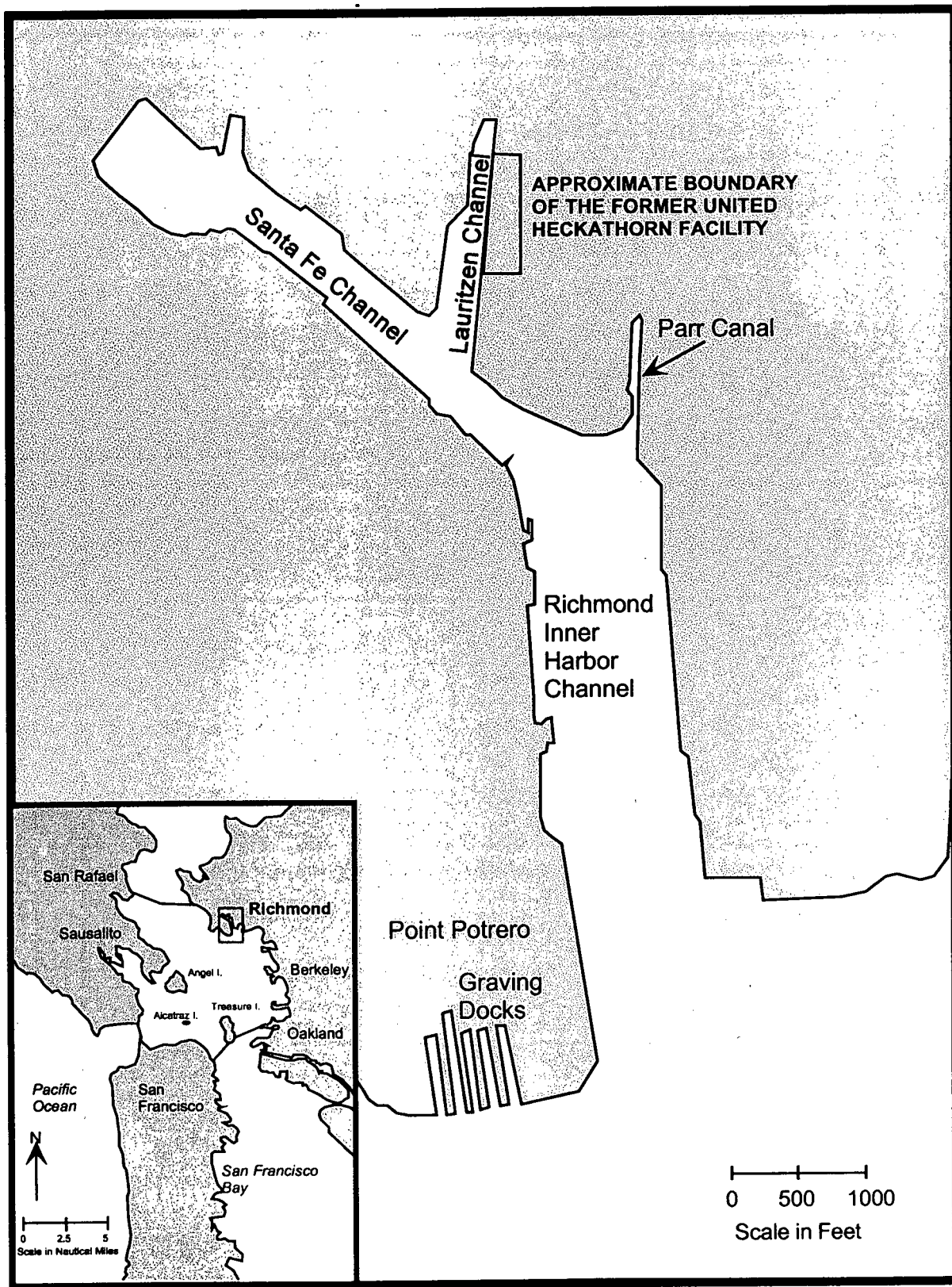


Figure 1.1. Location of the United Heckathorn Superfund Site, Richmond, California.

Remediation levels protective of the environment and human health were established to provide benchmarks for determining the effectiveness of the remediation actions. The Feasibility Study (Lincoff et al. 1994) and the ROD reviewed federal and state environmental laws that contained Applicable or Relevant and Appropriate Requirements (ARARs) for the remediation actions. EPA marine chronic and human health water quality criteria were identified as ARARs for surface water. Human health standards based on consumption of contaminated fish were used to establish remediation goals because they are lower than marine chronic criteria. No chemical-specific ARARs were identified as remediation goals for marine sediments or tissues at the site.

Sediment remediation by dredging, dewatering, and offsite disposal took place between July 1996 and March 1997. Extensive coring was conducted to verify that the younger-bay (contaminated) mud was removed and that only older-bay (less contaminated) mud remained. EPA collected post-remediation samples of the remaining older-bay mud, and analyses determined the average concentration of DDT to be 263  $\mu\text{g/kg}$  dry weight (Lincoff 1997), below the remediation goal of 590  $\mu\text{g/kg}$  DDT dry weight specified in the ROD. In April 1997, 9100 cubic yards of clean sand were placed in Lauritzen Channel to improve the older-bay mud surface for colonization by benthic invertebrates. The volume of sand was equivalent to an average depth of 1 ft over the dredged area, although the exact layer thickness undoubtedly varied because of the uneven, sloping channel bottom. Since remediation and sand placement in 1997, Lauritzen Channel has returned to industrial use by Levin Richmond Terminals and Manson Construction, resulting in frequent vessel traffic throughout the channel.

The purpose of the marine monitoring study is to document the expected reduction in flux of contaminants from the United Heckathorn Superfund Site following EPA response actions. The measurement endpoints for this long-term monitoring are mussels and surface waters. The remediation levels for waters set forth in the ROD are 0.59 ng/L for total DDT [the sum of the 4,4'- and 2,4'-isomers of DDT, DDD (TDE), and DDE] and 0.14 ng/L for dieldrin.

The first round (Year 1) of post-remediation biomonitoring was conducted six months after remediation (Antrim and Kohn 2000a). Year 1 biomonitoring showed that pesticide concentrations in the tissues of mussels exposed at the site were higher than those observed before remediation. Year 2 monitoring, conducted about 18 months after remediation, showed tissue levels that were much reduced from Year 1 and that only exceeded preremediation levels at Richmond Inner Harbor Channel (Antrim and Kohn 2000b). During both years the concentrations were higher at Lauritzen Channel stations than at the

Richmond Inner Harbor Channel or Santa Fe Channel stations. These results suggested that DDT was still present and bioavailable in Lauritzen Channel, especially near its head.

This report focuses on the Year 3 (2000) post-remediation biomonitoring results. Year 3 biomonitoring repeated the water and resident mussel tissue sampling and analyses of Years 1 and 2 (1997–1999). In contrast to previous years, EPA decided not to measure transplanted mussels for post-remediation monitoring in Year 3 (Appendix A). Year 3 results are compared with water and tissue pesticide data from two preremediation studies (Lee et al. 1994, Rasmussen 1995) and the Years 1 and 2 monitoring studies (Antrim and Kohn 2000a, b). Comparisons with Years 1 and 2 were done using the revised data for those years, published in 2000; the reports published in 1998 and 1999 reported tissue data with incorrect units (dry weight instead of wet weight) and therefore required correction. Corrected copies of the Year 1 and Year 2 monitoring reports are available on the web at <http://www.pnl.gov/main/publications>. Mussel tissue samples were collected and analyzed in both preremediation studies, but water samples were analyzed only for the ecological risk assessment (Lee et al 1994). The four post-remediation water and tissue monitoring stations are the same as the State Mussel Watch Program stations in the project area.

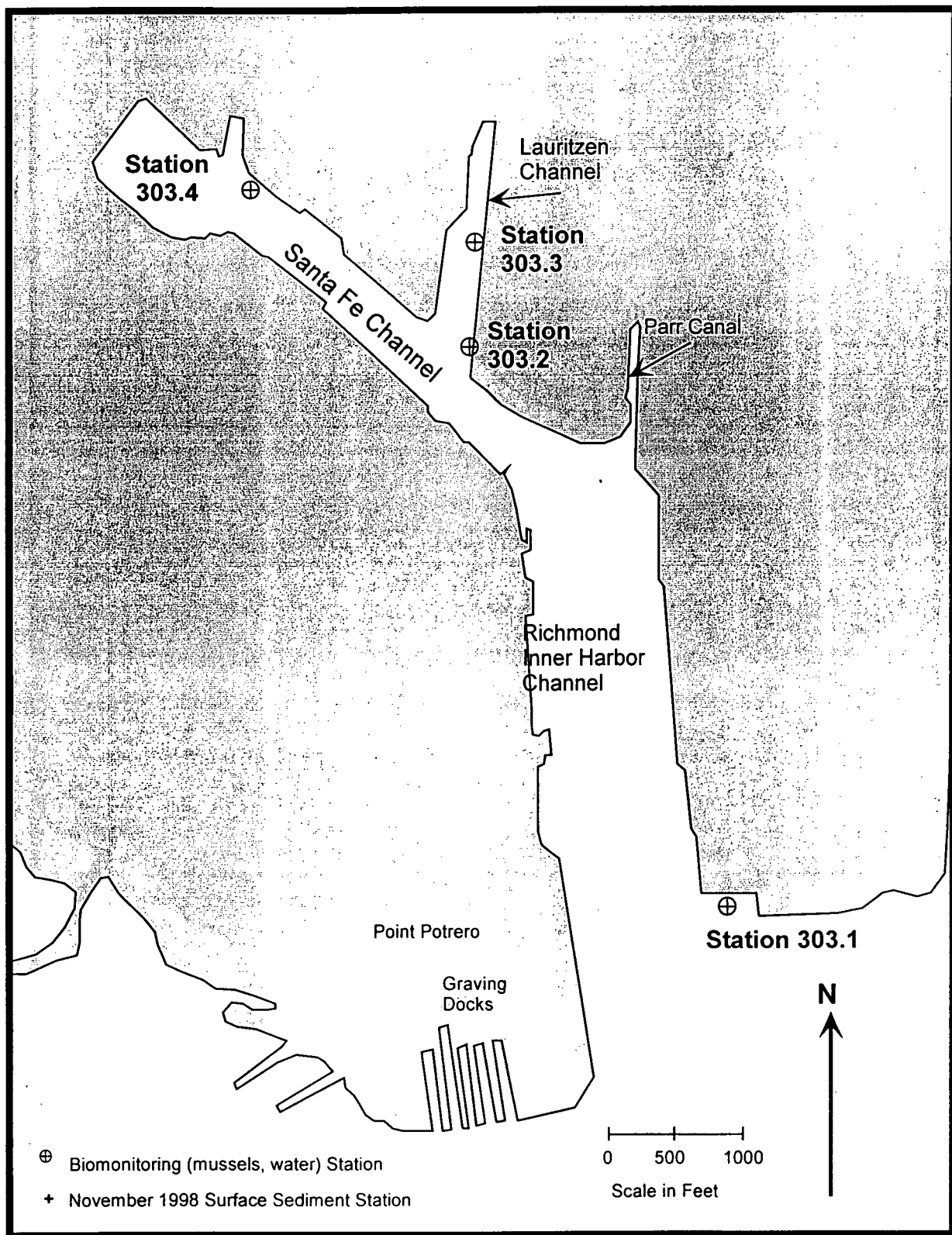
## 2.0 METHODS

Detailed methods for the collection, processing, and analysis of tissue and water samples in Year 3 were outlined in the Field Sampling and Analysis Plan (Battelle 1997) and were the same as those used in Years 1 and 2 post-remediation monitoring. A brief review of these methods is provided here. All procedures for sampling, sample custody, field and lab documentation, other aspects of documentation, quality assurance, and sample analysis were consistent with the more general procedures described in the Quality Assurance Project Plan (QAPP) for Remediation Investigation and Feasibility Study of Marine Sediments at the United Heckathorn Superfund Site (Battelle 1992). All samples were collected by EPA and analyzed at Battelle Marine Sciences Laboratory (MSL).

The four post-remediation monitoring stations selected are those stations in the project area that were sampled during the State Mussel Watch Program (Figure 2.1). Three of the stations also approximate locations sampled during the Ecological risk assessment (Lee et al. 1994). The Lauritzen Channel/End Station (Mussel Watch Station 303.3) corresponds to the Ecological Risk Assessment-Lauritzen Channel Station; the Santa Fe Channel Station (Mussel Watch Station 303.4) corresponds to the Ecological Risk Assessment-Santa Fe Channel Station. The Richmond Inner Harbor Channel Station (Mussel Watch Station 303.1) is approximately 1200 ft inshore from the Ecological Risk Assessment-Richmond Inner Harbor station, which was at navigational nun buoy (No. 16). The Ecological risk assessment had no sampling station near the entrance to Lauritzen Channel (Mussel Watch Station 303.2, Lauritzen Channel/Mouth). A more detailed description of sampling stations for the Year 3 biomonitoring is provided in Table 2.1 and in the Field Sampling Summary and Field Sampling Report memo (Appendix A).

### 2.1 TISSUE AND WATER SAMPLE COLLECTION

Approximately 45 resident blue mussels (*Mytilus edulis*) were collected from each of the four stations on February 15, 2000 (Figure 2.1). Resident mussels could have been one of several subspecies or hybrids in the *M. edulis* complex that cannot easily be distinguished by the shells alone (Harbo 1997). The coordinates presented in Table 2.1 for each station were determined in 1998 by using a Global Positioning System with differential correction. In Year 3, stations were revisited by using the visual landmarks listed in Table 2.1. Mussels were collected near the surface of the water, at about mean lower low water (MLLW) at Richmond Inner Harbor Channel (Station 303.1) and -0.4 ft MLLW at Lauritzen Channel/Mouth and Lauritzen Channel/End (Stations 303.2 and 303.3, respectively). At Santa Fe



**Figure 2.1.** Sampling stations for long-term post-remediation monitoring of the United Heckathorn Site.

**Table 2.1. Sampling Stations for Year 3 Post-remediation Monitoring (1999–2000) of the United Heckathorn Site**

Station Number	Station Name	Location <sup>(a)</sup>	Landmarks
303.1	Richmond Inner Harbor Channel	37°54' 32.8" N 122°21' 34.5" W	On western most wooden dolphin, near abandoned Ford automotive plant, southeast of public fishing pier
303.2	Lauritzen Channel/Mouth (South)	37°55' 12.6" N 122°22' 01.2" W	On east side of canal, on pilings beneath the Levin Dock near the northern end of a large wooden fender structure
303.3	Lauritzen Channel/End (North)	37°55' 22.5" N 122°21' 59.9" W	On east side of canal, southern end of small wooden pier that extends out into the channel
303.4	Santa Fe Channel/End	37°55' 21.53" N 122°21' 18.37" W	At northwest corner of floating boat shed, east of small boat fuel dock

(a) Data from January 6, 1998.

Channel/End (Station 303.4), mussels were collected near the surface from a floating dock. Thus, mussels at the Santa Fe Channel/End station were at a fixed depth relative to the water surface. Weather at the time of collection was calm with high clouds. Ambient water temperature was 12°C. During the time of collection an oily sheen was present on the water surface at all stations. There was heavy tug and barge traffic at all stations except Richmond Inner Harbor Channel. At the Lauritzen Channel/End station, tugboat operations caused a current estimated at several knots. High resuspension and mixing of bottom sediments was observed there, as noted in the field sampling report prepared by EPA Region 9 (Appendix A). Because of this resuspension, water samples collected from Lauritzen Channel/End were extremely turbid.

Mussels were cleaned gently in the field to remove external growth and packaged whole in ashed foil and plastic bags. Mussels were frozen at -20°C, shipped to the analytical laboratory in coolers, and held at -20°C until they were prepared for analysis. To prepare tissue samples, mussels were partially thawed, the valve or shell length was measured, byssal threads were cut from the tissue, and soft tissues were transferred to a sample jar. Sand and mud on the soft tissue were rinsed off with deionized water. Each

tissue sample consisted of from 42 to 46 mussels. The total wet weight of each tissue sample was recorded. Tissue samples were refrozen and stored at -20°C until extracted.

On February 15, 2000, surface water samples were collected approximately 1 ft (0.3 m) below the water surface. To collect a sample, a bottle was submerged, the cap was removed underwater to allow water in, and the cap replaced before the bottle was lifted from the water. At each station, three 2-L water samples were collected for analysis. Additional water samples were collected for quality control (i.e., matrix spike, matrix spike duplicate, and blind duplicate samples) analyses. Water samples were chilled to and held at 4°C until extracted. Salinity of the water samples was not measured in the field or in the laboratory.

## **2.2 TISSUE AND WATER SAMPLE ANALYSIS**

Chemical analyses followed methods described in the QAPP (Battelle 1992). The water and tissue samples collected on February 15 were extracted (February 18-22 for water; March 1 for tissue) and analyzed for chlorinated pesticides and PCB aroclors (March 21) within acceptable holding times. Tissue samples were also analyzed for percent lipids. Achieved detection limits in water and tissue samples determined by previous studies at MSL and the sample volume (water) or weight (tissues) were used to calculate sample-specific detection limits (Appendix B). Total DDT was calculated as the sum of detected concentrations for six DDT compounds: 2,4-DDE, 4,4-DDE, 2,4-DDD, 4,4-DDD, 2,4-DDT, and 4,4-DDT. The calculation of total DDT followed the California State Mussel Watch Program (Rasmussen 1995) and the ecological risk assessment for the United Heckathorn Superfund Site (Lee et al. 1994) methods that did not include sample data below the detection limits.

### 3.0 RESULTS AND DISCUSSION

This section presents the results of physical measurements to assess the size and condition of the resident mussels, and the results of chemical analyses of the water and mussel tissue samples. All extractions and analyses were conducted within the target holding times specified in the QAPP. Complete data tables, including QC data, are provided in Appendix B. In the following discussion, the Year 3 water data are compared to preremediation data from the ecological risk assessment, post-remediation data from 1998 and 1999, and the remediation goals for the site. The Year 3 tissue data are compared to preremediation tissue concentrations from the California State Mussel Watch Program and the ecological risk assessment, and to post-remediation data from 1998 and 1999.

#### 3.1 MUSSEL SIZE AND CONDITION

Raw data for shell-length measurements and mean wet weight per mussel are provided in Appendix C. Only resident (i.e., naturally-occurring) mussels were analyzed. Mussels collected for tissue samples ranged from 3.0 cm to 7.7 cm in shell length (Table 3.1). Shell lengths of 29 mussels (~16% of the total) were not within the preferred size range of 4.0 to 6.5 cm, which is a combination of the preference ranges cited by Rasmussen (1995) and Lee et al. (1994). The station mean wet weight per mussel, which was calculated as the total wet weight of the station tissue sample divided by the number of individuals per sample, ranged from 3.3 to 8.2 g (Table 3.1). The overall mean wet weight per mussel (calculated as the mean of the station means) was 5.34 g.

Lipid content of resident mussels ranged from 7.87% to 9.73% dry weight (Table 3.1; grand mean = 8.78; standard deviation = 0.93). Note that tissue lipid content is not a definitive indicator of organism health, because lipid content in bivalves can vary significantly depending on the availability of food and the bivalve's reproductive cycle. However, because nonpolar organic contaminants tend to accumulate in fatty tissues, normalizing contaminant data to mussel lipid content permits more equitable comparisons among samples to be made.

**Table 3.1. Summary of Length and Weight Data from Mussels Collected for Tissue Samples in February 2000 for Post-remediation Monitoring of the United Heckathorn Superfund Site**

	<u>Station</u>			
	303.1 Richmond Inner Harbor Channel	303.2 Lauritzen Channel/Mouth	303.3 Lauritzen Channel/End	303.4 Santa Fe Channel/End
<u>Shell Length (cm)</u>				
n	46	45	42	44
min	3.6	3.6	4.4	3.0
max	6.6	7.1	7.7	7.0
mean	4.93	5.34	5.74	4.87
standard deviation	0.74	0.95	0.82	0.82
n outside range <sup>(a)</sup>	4	10	11	4
grand mean	4.87			
standard deviation	3.77			
<u>Tissue Wet Weight (g)</u>				
sample weight	151.95	276.78	345.39	161.20
mean wt/mussel	3.30	6.15	8.22	3.66
grand mean	5.34			
standard deviation	2.30			
<u>Lipid Content (% dry weight)</u>				
	9.41	9.73	8.09	7.87
grand mean	8.78			
standard deviation	0.93			
<u>(a) number of individuals outside preferred range (4.0-6.5 cm)</u>				

### 3.2 WATER

The triplicate water samples that were collected at each site only provide short-term information about the water-column concentrations of DDT compounds and dieldrin. Such data, however, provide no information about the temporal variability or vertical stratification of these contaminants in the water column, information that would be useful in the interpretation of the biomonitoring results. The inability to evaluate temporal or spatial variability of water chemistry should be considered when these data are compared with the results of earlier studies. The differences between two such sampling events do not necessarily verify trends; nor are individual samples necessarily representative of typical conditions.

Recoveries of spiked surrogate compounds (PCB 103 and PCB 198) in Year 3 water samples ranged from 36.2% to 105%. Surrogate recovery for only one replicate (recovery = 36.2%, Station 303.2, Replicate c) was outside the target range (40%–120%). An individual compound's concentration in a sample was corrected according to the sample-specific surrogate recovery of the spiked compound (either PCB103 or PCB198) that elutes at a similar time on the chromatogram. Blank spike recoveries of dieldrin and 4,4'-DDT were within the target range (40%–120%) except for 4,4'-DDT in one blank sample (143%). In the method blank, two analytes were detected, 4,4'-DDE (0.10 ng/L ) and 4,4'-DDT (0.13 ng/L); samples with less than five times the blank concentration are flagged with a "B" in Table 3.2. Matrix spike levels for pesticides were not appropriate for the concentrations of the compounds occurring in the field samples. Matrix spike recovery for Aroclor 1254 was outside the target range (40%–120%) for one replicate (223%) and could not be calculated for the other replicate. Surrogate compound and blank spike recoveries indicated acceptable laboratory precision of the laboratory analyses.

Concentrations of total DDT in replicate water samples collected in Year 3 ranged from about 1.9 ng/L to 5152 ng/L (Table 3.2). Results were fairly consistent between replicates except at Station 303.3, which had one replicate with concentrations approximately ten times higher than the other replicates. The mean concentrations in Table 3.3 and Figure 3.1 are shown with and without the anomalous replicate. The high variability in replicate samples at Station 303.3 indicates that contaminants could be inconsistently distributed in the water column, perhaps in association with organic or particulate materials. Part of the variability is probably attributable to the resuspension of bottom sediments; field observations noted substantial vessel activity in the area and the presence of very turbid waters during the collection period.

With or without the anomalous replicate, Lauritzen Channel/End (Station 303.3) had the highest mean concentration of total DDT in 2000 (Table 3.3); the lowest mean concentration was from the Richmond Inner Harbor Channel (Station 303.1). Total DDT concentrations in Lauritzen Channel water were similar to or higher than those measured in 1999 (Figure 3.1; Table 3.4). In contrast, concentrations of total DDT in water from Richmond Inner Harbor Channel (Station 303.1) and Santa Fe Channel/End (Station 303.4) were lower in 2000 than in 1999 (Figure 3.1; Table 3.4). Concentrations of dieldrin in replicate water samples collected in Year 3 ranged from about 1.5 ng/L to 1710 ng/L (Table 3.2). Mean water-column concentrations of dieldrin ranged from 1.57 ng/L to 83 ng/L (Table 3.3; Station 303.3 mean calculated without replicate b). Although dieldrin was higher at all four stations in 2000 than in 1999, 2000 concentrations were similar to 1998 and preremediation concentrations (Figure 3.2; Table 3.4).

**Table 3.2.** Concentrations of DDT and Dieldrin in Water Samples Collected in February 2000 for Post-remediation Monitoring of the United Heckathorn Superfund Site

Station	Replicate	Location	Concentration (ng/L)								Aroclor 1254
			Dieldrin	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT	4,4'-DDT	Total DDT	
303.1	a	Richmond Inner Harbor Channel	1.65	0.37	0.36 B	0.38	1.03	0.20	0.64 B	3.0	13.3 U
303.1	b		1.61	0.24	0.24 B	0.27	1.04	0.26	0.72	2.8	14.6 U
303.1	c		1.45	0.01 U	0.41 B	0.02 U	0.77	0.29	0.43 B	1.9	12.7 U
303.2	a	Lauritzen Channel/ Mouth	10.50	0.17	1.23	4.66	17.2	0.92	3.86	28.0	18.0
303.2	b		8.60	0.15	1.49	4.00	16.0	1.40	5.85	28.9	21.7
303.2	c		7.79	0.31	1.59	4.16	14.1	1.56	5.17	26.9	25.6
303.3	a	Lauritzen Channel/ End	100	0.34	0.01 U	15.5	41.1	8.28	17.5	82.7	45.5
303.3	b*		1710 *	13 *	124 *	223 *	680 *	872 *	3240 *	5152 *	449 *
303.3	c		66	0.30	2.76	14.8	45.7	4.49	16.7	84.8	29.8
303.4	a	Santa Fe Channel End	2.68	0.07	0.46	0.64	1.99	0.37	1.38	4.9	13.6 U
303.4	b		2.16	0.01	0.39	0.58	1.39	0.29	0.82	3.5	14.1 U
303.4	c		1.50	0.09	0.39	0.40	1.03	0.23	0.56	2.7	12.7 U

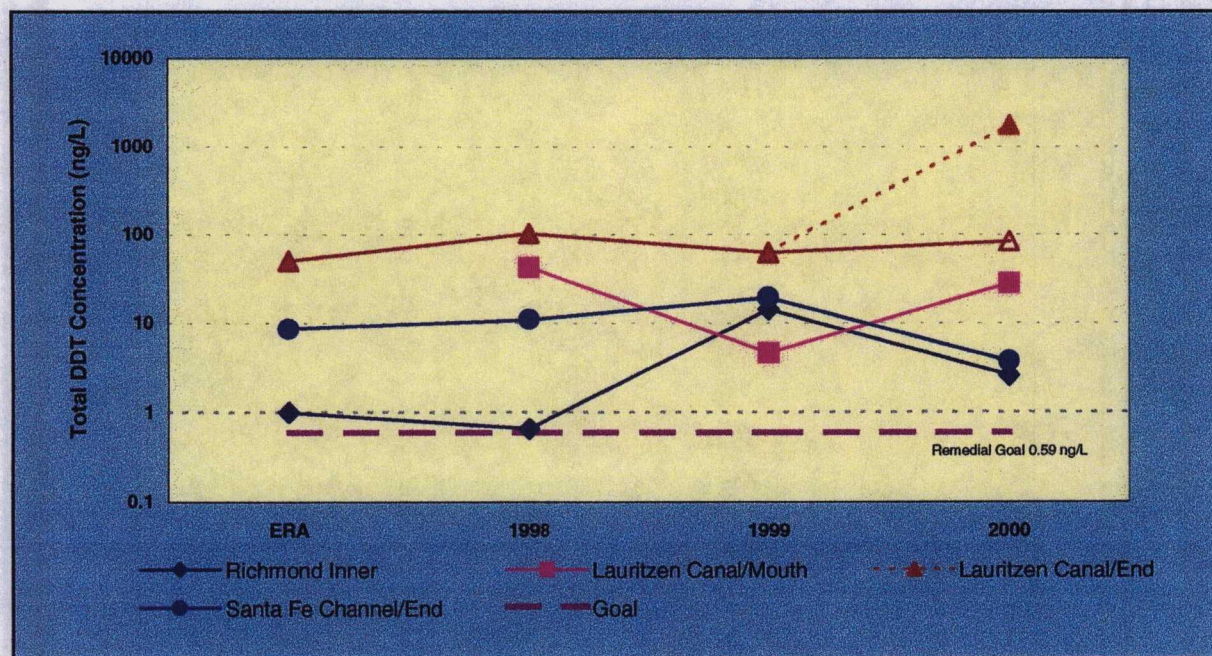
B Analyte detected in blank; concentration is less than 5 X blank value.

U Not detected at or above concentration shown.

\* 303.3 Replicate b was probably affected by sediment suspended in the water column due to vessel activity.

**Table 3.3.** Mean and Standard Deviation, (sd) Concentrations of DDT and Dieldrin in Water Samples Collected in February 2000 for Post-remediation Monitoring of the United Heckathorn Site

Station	Location	All Replicates		Excluding 303.3 Replicate "b"		
		Dieldrin ng/L	Total DDT ng/L	Dieldrin ng/L	Total DDT ng/L	
303.1	Richmond Inner Harbor Channel	1.6 0.11	2.6 0.56	1.6 0.11	2.6 0.56	Mean sd
303.2	Lauritzen Channel/ Mouth	9.0 1.39	27.9 1.00	9.0 1.39	27.9 1.00	Mean sd
303.3	Lauritzen Channel/ End	625.3 939.5	1773.2 2926.2	83.0 --	83.7 --	Mean sd
303.4	Santa Fe Channel/ End	2.1 0.59	3.7 1.12	2.1 0.59	3.7 1.12	Mean sd

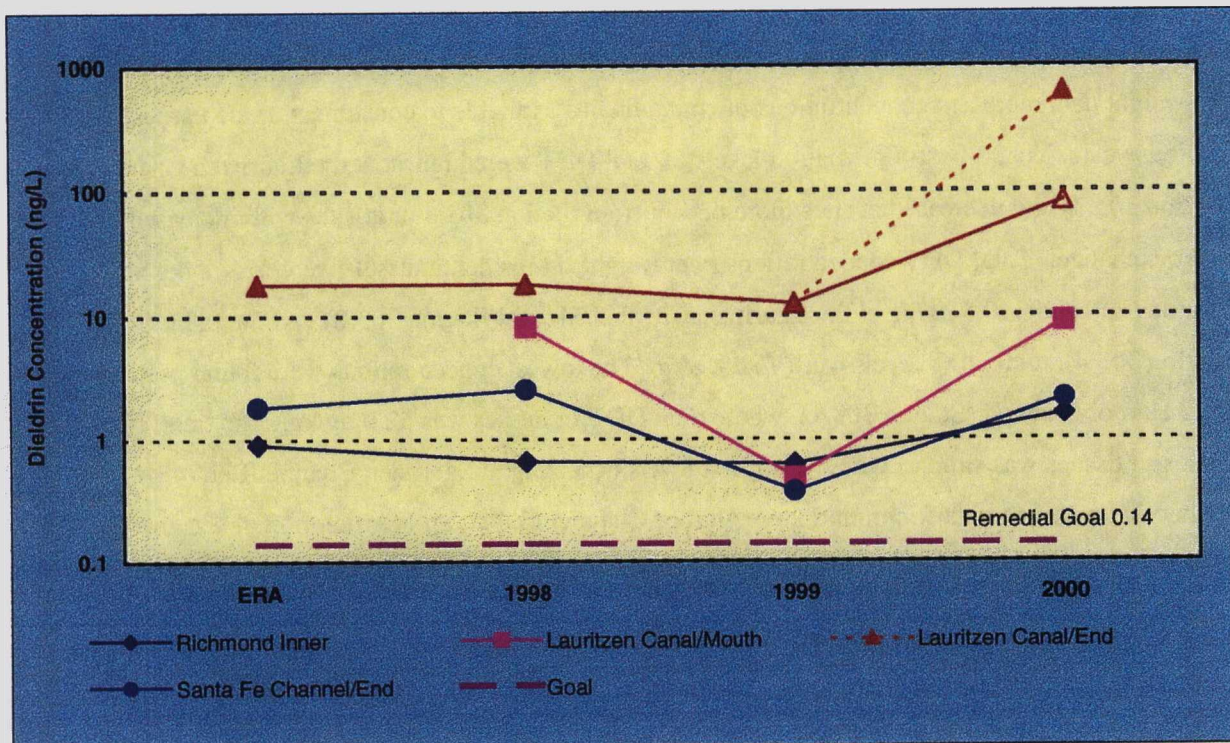


**Figure 3.1.** Comparison of preremediation (ecological risk assessment) and post-remediation total DDT concentrations in water samples collected at the United Heckathorn Site. The open triangle for station 303.3 is the mean value of only replicates a and c.

**Table 3.4. Comparison of Post-Remediation Concentration of Total DDT and Dieldrin in Water Samples with Preremediation Levels and Remedial Goal Concentrations**

Water		Water Concentration (ng/L)				
Sample ID	Location	Remediation Goal	Pre-Remediation <sup>(a)</sup>	1998 Post-Remediation	1999 Post-Remediation	2000 Post-Remediation
<u>Total DDT</u>						
303.1	Richmond Inner Harbor Channel	0.59	1	0.65	14.4	2.56
303.2	Lauritzen Channel/Mouth	0.59	no sample	42.6	4.61	27.9
303.3	Lauritzen Channel/End	0.59	50	103	62.3	83.7 (w/o rep b) 1773 (all reps)
303.4	Santa Fe Channel/End	0.59	8.6	11	19.2	3.70
<u>Dieldrin</u>						
303.1	Richmond Inner Harbor Channel	0.14	<1	0.65	0.62	1.57
303.2	Lauritzen Channel/Mouth	0.14	no sample	8.18	0.48	8.96
303.3	Lauritzen Channel/End	0.14	18	18.1	12.5	83 (w/o rep b) 625 (all reps)
303.4	Santa Fe Channel/End	0.14	1.8	2.47	0.37	2.11

(a) Pre-remediation water concentration is average of samples collected in October 1991 and February 1992 for the Ecological Risk Assessment (Lee et al. 1994)



**Figure 3.2.** Comparison of preremediation (ecological risk assessment) and post-remediation dieldrin concentrations in water samples collected at the United Heckathorn Site. The open triangle for station 303.3 is the mean value of only replicates a and c.

Water concentrations of total DDT and dieldrin were above remediation goals in all water samples and at all stations (Table 3.4, Figures 3.1 and 3.2). The most elevated contaminant concentrations are still found in Lauritzen Channel/End water (Station 303.3), where contaminated sediment remains and is periodically resuspended by vessel traffic. The variability shown between years at some stations and between replicates in 2000 for Station 303.3, highlight the statement made above that post-remediation water samples represent a “snapshot” of contaminant concentrations taken at a single point in time. Replicate variability and suspended sediment influence could be addressed in the future by analyzing both dissolved and total pesticides and PCBs in water samples, as well as total suspended solids.

### 3.3 TISSUES

Tissue samples from biomonitoring organisms provide a time-integrated indication of contaminant concentrations in the water column and are not as susceptible to small-scale temporal or spatial variability in contaminant concentrations as are water samples. For tissue analyses, all quality-control requirements, except the percent recovery of 4,4'-DDT from spiked blanks (122% and 127%), were met.

The post-remediation tissue data are summarized in Table 3.5 and compared with preremediation data in Tables 3.6 and 3.7. Evaluation of wet-weight data is appropriate for ecological risk assessment because wet-weight data represent concentrations of contaminants available to consumers of the tissues. As in previous years, Year 3 post-remediation levels of total DDT were highest at the Lauritzen Channel/End (Station 303.3) and decreased at sites more distant from Station 303.3 or at sites with increased exposure to water exchange. Total DDT concentrations (wet weight) in resident mussels were 522  $\mu\text{g/kg}$  at Lauritzen Channel/End and 310.5  $\mu\text{g/kg}$  at the Lauritzen Channel/Mouth (Station 303.2). At Santa Fe Channel/End (Station 303.4), total DDT levels were 75.2  $\mu\text{g/kg}$ . The lowest concentrations were found at Richmond Inner Harbor Channel (Station 303.1), where total DDT in tissues was 52.0  $\mu\text{g/kg}$ . The trend for dieldrin in mussel tissues was similar, with the highest levels occurring at Lauritzen Channel/End (42.7  $\mu\text{g/kg}$ ) and the lowest levels found at Richmond Inner Harbor Channel (5.4  $\mu\text{g/kg}$ ). Aroclor 1254 was the only PCB detected in mussels collected from post-remediation monitoring stations in 2000. Wet-weight PCB concentrations were highest in Lauritzen Channel/Mouth (187  $\mu\text{g/kg}$ ), and lowest at Santa Fe Channel/End (123  $\mu\text{g/kg}$ ) (Table 3.5).

Tissue contaminant burdens from Year 3 of post-remediation biomonitoring were very similar to Year 2 post-remediation levels (Table 3.6, Figure 3.3). Total DDT and dieldrin levels have shown very similar patterns of fluctuation in levels over the three years of post-remediation monitoring (Figures 3.3 and 3.4). In Year 1, total DDT (wet weight) was up to 3 times greater than the preremediation levels (Figure 3.3). Year 2 post-remediation biomonitoring levels were substantially reduced from the 1992 preremediation levels. Year 3 (2000) values were similar to but slightly less than (Stations 303.1 and 303.3) or slightly greater than (Stations 303.2 and 303.4) Year 2 levels.

The pattern for dieldrin was similar, as Year 1 (1998) post-remediation resident mussel tissue levels were greater than preremediation levels measured in 1992 (Lee et al. 1994) and Year 2 levels showed a substantive reduction from Year 1 levels (Figure 3.4). However, levels found in Year 3 were 1.5 to 3 times higher than Year 2 levels (Figure 3.4) and in one case (Station 303.1) were about the same as Year 1 levels.

The reduction in transplanted mussel tissue burdens of PCBs from preremediation to Year 2 (PCBs were not measured in Year 1) was substantial (Antrim and Kohn 2000b). Tissue concentrations of Aroclor 1254 (lipid-normalized) in Year 2 resident mussels (*M. edulis*) were higher than those for Year 2 transplanted mussels (*M. californianus*). However, PCBs in Year 2 resident mussels were still lower (29% to 77%; average 54%) than 1988 or 1991 (Mussel Watch) preremediation levels for transplanted mussels.

**Table 3.5.** Concentrations of DDT, Dieldrin, and PCB Aroclor 1254 in Tissue Samples Collected in February 2000 for Post-Remediation Monitoring of the United Heckathorn Site

Analyte	Station Location	Sample ID and Concentration ( $\mu\text{g/kg}$ )			
		303.1 Richmond Inner Harbor Channel	303.2 Lauritzen Channel Mouth	303.3 Lauritzen Channel End	303.4 Santa Fe Channel End
2,4'-DDD		4.9	38.6 D	60.5 D	7.0
2,4'-DDE		0.8	3.2	4.5	0.8
2,4'-DDT		4.0	34.5 D	83.5 D	7.1
4,4'-DDD		17.7	104.0 D	157.0 D	23.2
4,4'-DDE		13.5	65.4	74.5 D	18.0
4,4'-DDT		11.1	64.8 D	142.0 D	19.0
<b>Total DDT (wet wt)</b>		<b>52.0</b>	<b>310.5</b>	<b>522.0</b>	<b>75.2</b>
<b>Dieldrin (wet wt)</b>		<b>5.4</b>	<b>27.7</b>	<b>42.7</b>	<b>6.4</b>
Percent Dry Wt		12.5	10.2	8.0	10.4
Total DDT (dry wt)		416	3044	6525	723
Dieldrin (dry wt)		43	272	534	61
Lipids (% dry wt)		9.41	9.73	8.09	7.87
DDT (ppb <sup>(b)</sup> lipid)		4423	31281	80657	9182
Dieldrin (ppb lipid)		457	2791	6598	779
Aroclor 1254 (wet wt)		150	187	169	123
Aroclor 1254 (dry wt)		1200	1833	2113	1183
Aroclor 1254 (ppb lipid)		1594	1922	2089	1563

(a) Total DDT is sum of detected 2,4- and 4,4- DDD, DDE, and DDT.

(b) ppb parts per billion ( $\mu\text{g}$  contaminant/kg lipid).

PCB tissue burdens in resident mussels increased slightly in Year 3, with Year 3 levels up to 3 times greater than their Year 2 counterparts on a wet weight basis (Table 3.6). The apparent increase was somewhat lower when differences in lipid content were accounted for: on a lipid-normalized basis, Year 3 tissue PCBs were about 1.4 to 2.1 times greater than Year 2 (Table 3.7). The increase in tissue Aroclor 1254 burden in Year 3 samples versus Year 2 samples was similar at all stations.

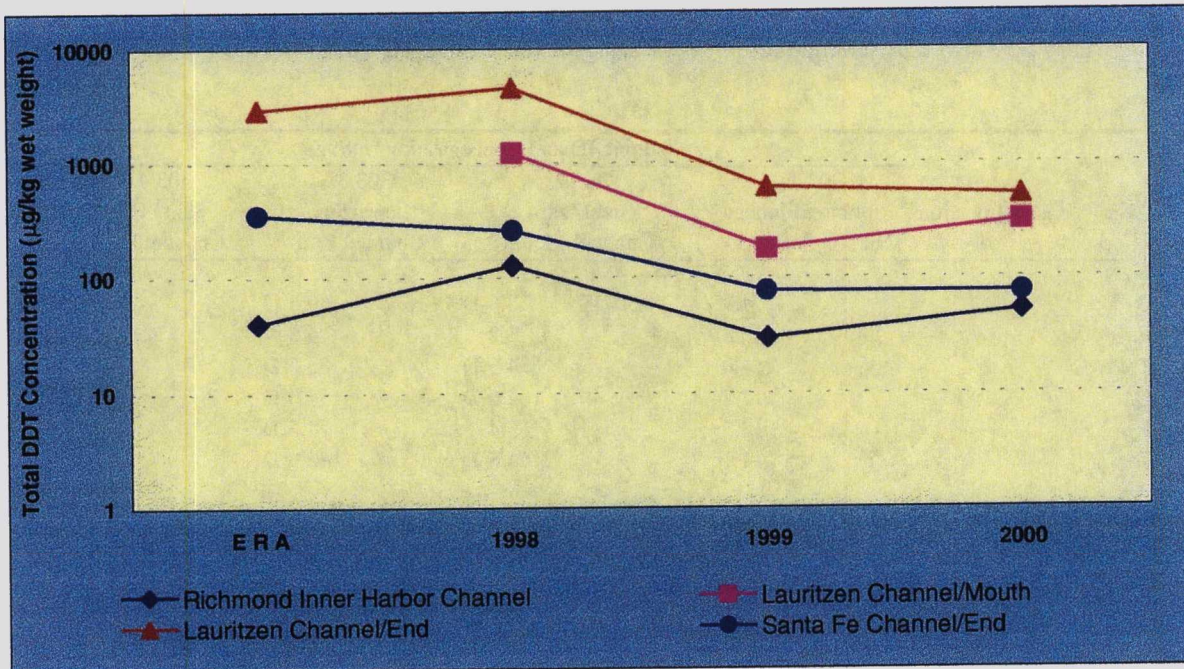


Figure 3.3. Comparison of preremediation (ecological risk assessment) and post-remediation total DDT concentrations in mussel tissue samples collected at the United Heckathorn Site.

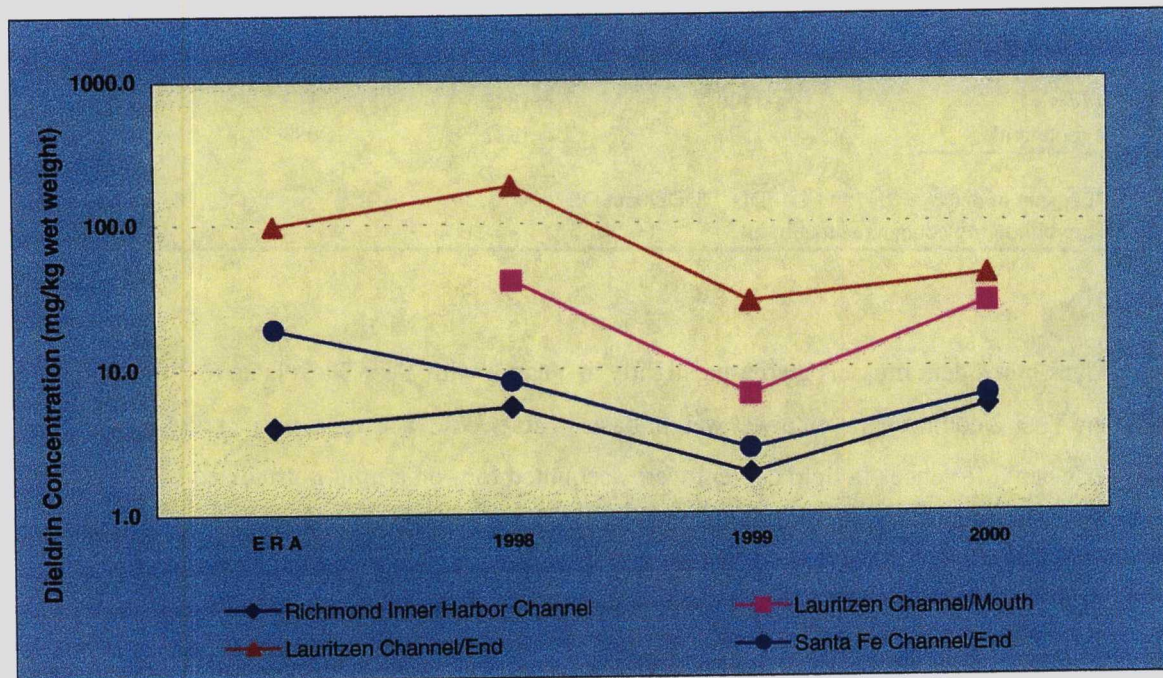


Figure 3.4. Comparison of preremediation (ecological risk assessment) and post-remediation dieldrin concentrations in mussel tissue samples collected at the United Heckathorn Site.

**Table 3.6.** Comparison of Post-Remediation Total DDT, Dieldrin, and PCBs in Tissues with Preremediation Concentrations ( $\mu\text{g/kg}$  wet weight)

Station Number	Station Name	State Mussel Watch <sup>(a)</sup>	Ecological Risk Assessment <sup>(b)</sup>	1998 (Year 1) Post-remediation	1999 (Year 2) Post-remediation	2000 (Year 3) Post-remediation
		Transplant	Resident	Resident	Resident	Resident
<b>Total DDT</b>						
303.1	Richmond Inner Harbor Channel	47.0 <sup>(c)</sup>	40	127	30	52
303.2	Lauritzen Canal/Mouth	629 <sup>(d)</sup>	---	1222	176	310
303.3	Lauritzen Canal/End	5074 <sup>(d)</sup> 1369 <sup>(c)</sup>	2900	4504	606	522
303.4	Santa Fe Channel/End	369 <sup>(c)</sup>	350	256	76	75
<b>Dieldrin</b>						
303.1	Richmond Inner Harbor Channel	7.7 <sup>(c)</sup>	4.0	5.43	1.9	5.4
303.2	Lauritzen Canal/Mouth	87.0 <sup>(d)</sup>	---	40.3	6.5	27.7
303.3	Lauritzen Canal/End	602 <sup>(d)</sup> 100 <sup>(c)</sup>	97.0	184	28.4	42.7
303.4	Santa Fe Channel/End	32.5 <sup>(c)</sup>	19.0	8.18	2.8	6.4
<b>Total PCBs</b>						
303.1	Richmond Inner Harbor Channel	176 <sup>(c)</sup>	not measured	not measured	51	150
303.2	Lauritzen Canal/Mouth	120 <sup>(d)</sup>	not measured	not measured	75	187
303.3	Lauritzen Canal/End	196 <sup>(d)</sup> 137 <sup>(c)</sup>	not measured	not measured	124	169
303.4	Santa Fe Channel/End	138 <sup>(c)</sup>	not measured	not measured	67	123
(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).						
(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).						
(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).						
(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).						

**Table 3.7.** Comparison of Lipid-Normalized Post-remediation Total DDT, Dieldrin, and PCBs in Tissues with Lipid-Normalized Preremediation Concentrations ( $\mu\text{g/kg}$  lipid)

Station Number	Station Name	State Mussel Watch <sup>(a)</sup>	Ecological Risk Assessment <sup>(b)</sup>	1998 (Year 1) Post-remediation	1999 (Year 2) Post-remediation	2000 (Year 3) Post-remediation
		Transplant	Resident	Resident	Resident	Resident
<b>Total DDT</b>						
303.1	Richmond Inner Harbor Channel	9,215 <sup>(c)</sup>	3,275	12,313	4,672	4,423
303.2	Lauritzen Channel/Mouth	78,481 <sup>(d)</sup>		134,633	24,855	31,281
303.3	Lauritzen Channel/End	583,819 <sup>(d)</sup> 380,361 <sup>(c)</sup>	250,411	427,423	94,061	80,657
303.4	Santa Fe Channel/End	47,283 <sup>(c)</sup>	21,919	45,695	8,193	9,182
<b>Dieldrin</b>						
303.1	Richmond Inner Harbor Channel	1,507 <sup>(c)</sup>	322	525	293	457
303.2	Lauritzen Canal/Mouth	10,861 <sup>(d)</sup>		4,439	919	2,791
303.3	Lauritzen Canal/End	69,272 <sup>(d)</sup> 27,778 <sup>(c)</sup>	8,590	17,463	4,410	6,598
303.4	Santa Fe Channel/End	4,167 <sup>(c)</sup>	1,126	1462	300	779
<b>Total PCBs</b>						
303.1	Richmond Inner Harbor Channel	34,440 <sup>(c)</sup>	not measured	not measured	8,020	12,752
303.2	Lauritzen Canal/Mouth	14,981 <sup>(d)</sup>	not measured	not measured	10,599	18,842
303.3	Lauritzen Canal/End	22,554 <sup>(d)</sup> 38,056 <sup>(c)</sup>	not measured	not measured	19,255	26,112
303.4	Santa Fe Channel/End	17,667 <sup>(c)</sup>	not measured	not measured	7,302	15,028

(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).

(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).

(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).

(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).

#### 4.0 CONCLUSIONS

Results from the third post-remediation monitoring survey indicated that chlorinated pesticides remained in the Lauritzen Channel and in the semi-enclosed waters nearby. Discrete water samples collected in February 2000 indicated that the total DDT and dieldrin concentrations in the water were similar to preremediation levels. Thus, remediation goals for total DDT and dieldrin in water have not yet been achieved for the study site. Year 3 biomonitoring showed that the bioavailability of total DDT and dieldrin, as demonstrated by concentrations in tissues from resident mussels, was lower at the Lauritzen Channel/End and Santa Fe Channel/End stations relative to preremediation data. Bioavailability of these two pesticides also decreased between Year 1 and Year 2 of biomonitoring, but was similar to Year 2 in Year 3. Tissue concentrations of the PCB Aroclor 1254 were much lower than Mussel Watch preremediation levels at Richmond Inner Harbor Channel, but were similar to or higher than Mussel Watch levels in the Lauritzen Channel and Santa Fe Channel/End. Biomonitoring using mussel tissues will continue to document changes in the long-term bioavailability of pesticides from the Lauritzen Channel sediment that cannot be assessed through water-sample analyses.

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**APPENDIX A**

**FIELD SUMMARY REPORT**

Field Sampling Summary for Mussels and Surface Water  
at the United Heckathorn Site in  
Richmond, California, conducted 2/15/2000.

Andrew Lincoff  
EPA Region 9 Laboratory  
PMD-2  
March 10, 2000

INTRODUCTION

This sampling event involved the collection of mussels and surface water samples from the Lauritzen Channel at the United Heckathorn Superfund Site and at other locations in Richmond Harbor in Richmond, California. Sampling was performed on February 15, 2000 by Andrew Lincoff and Mark Petersen of the EPA Region 9 Laboratory. Sampling was performed in accordance with Battelle's "United Heckathorn Post-Remediation Field Monitoring Plan" (FSP), dated February 5, 1997.

OBJECTIVE

EPA conducted this field sampling as part of the oversight of a final Remedial Action under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) at the United Heckathorn Site in Richmond, California. The sampling effort involved collecting physical environmental samples to analyze for the presence of hazardous substances.

The United Heckathorn Site was used to formulate pesticides from approximately 1947 to 1966. Soils at the Site and sediments in Richmond Harbor were contaminated with various chlorinated pesticides, primarily DDT, as a result of these pesticide formulation activities. The final remedy contained in EPA's October, 1994 Record of Decision addressed remaining hazardous substances, primarily in the marine environment. The major marine components of the selected remedy included:

- Dredging of all soft bay mud from the Lauritzen Channel and Parr Canal, with offsite disposal of dredged material.
- Marine monitoring to verify the effectiveness of the remedy.

The first component of the remedy selected in the ROD called for dredging all "young bay mud" from those channels in Richmond Harbor which contained average DDT concentrations greater than 590 ppb (dry wt.). The dredging was completed in April, 1997. The short-term monitoring, performed according to EPA's September 5, 1996 FSP, consisted of sediment chemistry monitoring to ensure that the average sediment concentration after dredging was below the cleanup level selected in the ROD. This monitoring was completed shortly prior to the placement of the sand layer in April, 1997.

Long-term monitoring is addressed by Battelle's February 5, 1997 FSP. The purpose of the long-term monitoring is to demonstrate the effectiveness of the remedy. Prior to the remediation, mussels in the Lauritzen Channel contained the highest levels of DDT and dieldrin in the State, and surface water exceeded EPA's Ambient Water Quality Criteria for DDT by a factor of 50. Lower but still elevated levels were found in mussels and surface water in the Santa Fe Channel. It was concluded in EPA's Remedial Investigation that these elevated levels were the result of continuous flux from contaminated sediments. Approximately 98% of the mass of DDT in sediments in Richmond Harbor was removed by the remedial dredging. The long-term monitoring will demonstrate whether this action has succeeded in reducing the levels of DDT in mussels and surface waters.

Battelle's FSP included monitoring using both transplanted California mussels and resident Bay mussels. The first round of the long-term sampling occurred in January, 1998. The second round occurred in March, 1999. This is the third round of sampling. The seasonal timing was chosen to match the protocol used by the California State Mussel Watch Program, in order to permit comparison with the State's results over the past 15 years. In the first two rounds, both transplanted and resident mussels are analyzed to determine any difference. Based on the results of the first two rounds and discussions with California State Mussel Watch Program personnel, only resident mussels were collected in the third round.

Laboratory results are expected from Battelle in approximately one month.

#### FIELD NOTES AND OBSERVATIONS

1. Samples were collected on February 15, 2000 at low tide. The weather during the sampling was calm with high clouds.

2. The sample station numbers, locations, date and times, and other information are listed in Table 1, below. Location coordinates were determined using GPS with differential correction on 1/6/98. As discussed in the FSP, the station numbers are those used by the California Mussel Watch Program. Station 303.1 is at the entrance to the Richmond Inner Harbor Channel near the old Ford automotive plant. Station 303.2 is on the eastern side of the Lauritzen near its mouth, beneath the Levin Dock near the northern end of a large wooden fender structure. Station 303.3 is approximately 2/3 of the way up the Lauritzen Channel, on the eastern side. Mussels were collected from the southern end of a small wooden pier which extends out into the channel. This location is very close to where the highest levels of pesticide residues were removed from the Heckathorn Site. Station 303.4 is in the upper Santa Fe Channel at the far western end of a large covered floating marina on the northern side. Due to boats tied up at this location, the mussels were collected near to the middle of the floating marina.

Table 1  
Mussel and Seawater Sample Locations

<u>Station</u>	<u>Date</u> <u>Time</u>	<u>Location</u>	<u>Remarks</u>
303.1	2/15/00 1357	37 54' 32.8" N Richmond Channel 122 21' 34.5" W	Blind Dup. Seawater labeled 303.5
303.2	2/15/00 1505	37 55' 12.6" N Lauritzen South 122 22' 01.2" W	
303.3	2/15/00 1435	37 55' 22.5" N Lauritzen North 122 21' 59.9" W	MSD Seawater
303.4	2/15/00 1415	37 55' 21.53" N 122 21' 18.37" W	Santa Fe

Seawater and resident Bay mussels were collected at each station for analysis by Battelle. At each station three 2 liter replicate seawater samples were collected. At station 303.3, two additional 2 liter seawater samples were collected for Battelle QA/QC. An additional single 2 liter blind duplicate of seawater sample 303.1 was collected and shipped to the Battelle Lab with the fictitious station number 303.5.

At each station, approximately 45 resident mussels were collected. The 45 mussels per sample sent to Battelle is large enough for any sample to be selected by Battelle for laboratory QA/QC.

The resident mussels were all collected near the surface, which at the collection times and dates was approximately at Mean Lower Low Water (MLLW) for the mussels collected from pilings at station 303.1, and -0.4 ft MLLW for stations 303.2, and 303.3. At station 303.4, the mussels were collected near the surface from a floating dock.

3. The water temperature at each station was 12 degrees C.

4. An oily sheen was present on the water at all stations.

5. There was heavy tug and barge traffic at all stations except 303.1. At station 303.3 tugboat operations caused a current estimated at several knots and high resuspension and mixing of bottom sediments. The water samples from 303.3 were extremely turbid due to the suspended sediments.

**APPENDIX B**

**ANALYTICAL RESULTS FROM  
WATER AND TISSUE SAMPLES**

## QA/QC SUMMARY

**PROJECT:** Heckathorn Biomonitoring Year 3  
**PARAMETER:** Pesticides, PCBs, and Total Lipids  
**LABORATORY:** Battelle/Marine Sciences Laboratory, Sequim, Washington  
**MATRIX:** Tissues

**SAMPLE CUSTODY:** Four mussel tissue samples were received on 2/17/00. All samples were received in good condition. The cooler temperature on arrival was 0.3°C. Mussels were shucked in the wet laboratory, placed in clean glass jars, and returned to the chemistry laboratory for analysis on 3/01/00. The temperature was not recorded; samples were hand-delivered. Mussel samples were then assigned a Battelle Central File (CF) identification number (1466) and were entered into Battelle's log-in system. One sample [1466-9] was received in two jars – the contents of both jars were combined before analysis.

### QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limits</u>	
					<u>Target (ng/g wet)</u>	<u>Achieved (ng/g)</u>
2,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.27
Dieldrin	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.29
4,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	1.03
2,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.38
4,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.36
2,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.52
4,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	2	0.36
PCB Aroclor 1242	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	20	14.3
PCB Aroclor 1248	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	20	14.3
PCB Aroclor 1254	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	20	14.3
PCB Aroclor 1260	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	20	14.3
Total Lipids	CHCl <sub>3</sub>	Gravimetric	NA	±30%	NA	NA

**METHOD:** Tissue samples for analysis of chlorinated pesticides and PCBs were processed according to Battelle SOP MSL-O-009, *Extraction and Clean-Up of Sediments and Tissues for Semivolatile Organics Following the Surrogate Internal Standard Method*, which is derived from NOAA NS&T and EPA methods with modifications from Krahn et al. (1988). Tissue samples were macerated and extracted with methylene chloride. Interferences were removed using an aluminum/silicon column chromatography step followed by a high-performance liquid chromatography (HPLC) clean-up according to SOP MSL-O-006, *HPLC Cleanup of Sediment and Tissue Extracts for Semivolatile Pollutants*. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column (DB-1701) gas chromatography with electron-capture detection (GC/ECD) according to SOP MSL-O-004, *Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection*, which is based on EPA Method 8081 (EPA 1986). Total lipids were determined according to the Bligh et al. (1959) method modified to use a smaller sample size. Lipids were extracted from separate aliquots of tissue samples using chloroform and methanol, and the lipid weight obtained gravimetrically.

## QA/QC SUMMARY

**HOLDING TIMES:** All extractions and analyses were conducted within target holding times: 14 days to extraction (refrigerated, not frozen), and 40 days to analysis after extraction. Samples were received on 3/1/00 and held at 4°C. Samples were extracted on 3/1/00 and analyzed on 3/21/00. Lipid extractions were conducted on 3/6/00.

**DETECTION LIMITS:** Detection limits were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.

Sample detection limits are calculated using the achieved detection limit and the sample weight.

**BLANKS/BLANK SPIKES:** One procedural blank and two blank spikes were analyzed. All spiked analytes (dieldrin, 4,4'-DDT, and PCB Aroclor 1254) were undetected in the blank. Blank spike recoveries of dieldrin and Aroclor 1254 were within the target range of 40%-120%. Blank spike recoveries of 4,4'-DDT slightly exceeded the target range at 122% and 127%.

**REPLICATES:** One tissue sample [1466-8 (20212-Y3M-03, Station 303.2)] was analyzed in duplicate for chlorinated compounds. Precision for duplicate analysis is reported by calculating the relative percent difference (RPD) of replicate results. RPDs for all analytes of interest ranged from 3% to 23%, and were all within the QC limits of  $\pm 30\%$ .

Sample [1466-7 (20212-Y3M-02, Station 303.4)] was analyzed in duplicate for lipids. Precision of the duplicate lipid analysis was within the QC limits of  $\pm 30\%$  (3%).

**MATRIX SPIKES:** A matrix spike and matrix spike duplicate pair was analyzed using sample 20212-Y3M-04 (Station 303.3). Recoveries of the three spiked analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were within the target range of 40%-120% in both the MS and MSD.

Replicate precision of the MS/MSD analysis, expressed as the RPD between the MS and MSD, was within the QC criteria of  $\pm 30\%$  for dieldrin (2%) and Aroclor 1254 (7%). Precision of the MS/MSD analysis for 4,4'-DDT (58% RPD) exceeded QC criteria. No corrective action was taken.

**SURROGATE RECOVERIES:** Chlorinated compounds PCBs 103 and 198 were added to each sample during the preparation step as surrogates to assess the efficiency of the extraction procedure. Surrogate recoveries were within the target range of 40%-120%, ranging from 64.0% to 84.5%.

## QA/QC SUMMARY

### REFERENCES:

Bligh, E.G., and W.J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. *Canadian Journal of Biochemistry and Physiology*. 37:8 911-917.

Krahn, M.M, CA Wigren, R.W. Pearce, S.K. Moore, R.G. Bogar, W. D. McLeod, Jr., S.L. Chan, and D.W. Brown. 1988. *New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants*. NOAA Technical Memorandum MNFS F/NWC-153. Standard Analytical Procedures of the NOAA National Facility, 1988. National Oceanic and Atmospheric Administration, National Marine Fisheries Service, Seattle, WA.

U.S. EPA. 1986 (Revised 1990). *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Tissues

Samples Received 3/1/00

MSL Code	1466-6	1466-7	1466-8	1466-9
STATION NO	20212-Y3M-01	20212-Y3M-02	20212-Y3M-03	20212-Y3M-04
LOCATION				
Matrix	Tissue	Tissue	Tissue	Tissue
Wet Wt (g)	10.0	10.1	10.1	10.7
Percent Wet Wt	87.5	89.6	89.8	92.0
Extraction Date	03/01/2000	03/01/2000	03/01/2000	03/01/2000
Percent Lipids (DW)	9.41	7.87	9.73	8.09
Dilution			10X	10X
Analytical Batch	1	1	1	1
Unit (wet wt)	ng/g	ng/g	ng/g	ng/g
2,4'-DDE	0.81	0.84	3.15	4.51
Dieldrin	5.38	6.38	27.7	42.7
4,4'-DDE	13.5	18.0	65.4	74.5 D
2,4'-DDD	4.92	7.00	38.6 D	60.5 D
4,4'-DDD	17.7	23.2	104 D	157 D
2,4'-DDT	3.99	7.11	34.5 D	83.5 D
4,4'-DDT	11.1	19.0	64.8 D	142 D
<b><u>SURROGATE RECOVERIES (%)</u></b>				
PCB103	62.9	70.3	64.0	82.5 D
PCB198	68.1	71.1	73.3	79.6 D
<b><u>AROCLORS</u></b>				
1242	14.3 U	14.1 U	14.2 U	13.5 U
1248	14.3 U	14.1 U	14.2 U	13.5 U
1254	150	123	187	169
1260	14.3 U	14.1 U	14.2 U	13.5 U

U Not detected at or above DL shown

D Diluted 10x

B.4

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**  
 Pesticides and PCBs in Tissues  
 Samples Received 3/1/00

STATION NO LOCATION	BSA				BSB				DUP	
	Blank	Blank	Spike	Percent	Blank	Spike	Percent		1466-7	1466-7
									20212-Y3M-02	20212-Y3M-02
		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD		RPD
Matrix	Tissue	Tissue			Tissue				Tissue	Tissue
Wet Wt (g)	NA	NA			NA				10.1	10.1
Percent Wet Wt	NA	NA			NA				89.6	89.6
Extraction Date	03/01/00	03/01/00			03/01/00				03/01/00	03/01/00
Percent Lipids (DW)	NA	NA			NA				7.87	8.10
Dilution										3%
Analytical Batch	1	1			1				1	1
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%	ng/g	ng/g
2,4'-DDE	0.27 U	0.27 U	NS	NA	0.27 U	NS	NA		0.84	NA
Dieldrin	0.29 U	8.37	10.0	84%	9.39	10.0	94%	11%	6.38	NA
4,4'-DDE	1.03 U	1.03 U	NS	NA	1.03 U	NS	NA		18.0	NA
2,4'-DDD	0.38 U	5.36	NS	NA	5.34	NS	NA		7.00	NA
4,4'-DDD	0.36 U	0.36 U	NS	NA	0.36 U	NS	NA		23.2	NA
2,4'-DDT	0.52 U	0.52 U	NS	NA	0.52 U	NS	NA		7.11	NA
4,4'-DDT	0.36 U	12.2	10.0	122% #	12.7	10.0	127% #	4%	19.0	NA

**SURROGATE RECOVERIES (%)**

PCB103	79.0	69.3			68.7				70.3	NA
PCB198	84.5	68.6			70.3				71.1	NA

**AROCLORS**

1242	14.3 U	14.3 U			14.3 U				14.1 U	NA
1248	14.3 U	14.3 U			14.3 U				14.1 U	NA
1254	14.3 U	115	100	115%	118	100	118%	3%	123	NA
1260	14.3 U	14.3 U			14.3 U				14.1 U	NA

U Not detected at or above DL shown

D Diluted 10x

# Outside QAQC recovery limits

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**UNITED HECKATHORN**  
 Pesticides and PCBs in Tissues  
 Samples Received 2/17/00

B.6

DUP				MSA				MSB			
MSL Code	1466-8	1466-8		1466-9	1466-9	Spike	Percent	1466-9	Spike	Percent	
STATION NO	20212-Y3M-03	20212-Y3M-03		20212-Y3M-04							
LOCATION			RPD		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Tissue	Tissue		Tissue	Tissue			Tissue			
Wet Wt (g)	10.1	10.1		10.7	10.6			10.5			
Percent Wet Wt	89.8	89.8		92.0	92.0			92.0			
Extraction Date	03/01/00	03/01/00		03/01/00	03/01/00			03/01/00			
Percent Lipids	9.73	9.73		8.09	8.09			8.09			
Dilution	10X	10X		10X	10X			10X			
Analytical Batch	1	1		1	1			1			
Unit (wet wt)	ng/g	ng/g	%	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%
2,4'-DDE	3.15	2.91	8%	4.51	4.60	NS	NA	4.79	NS	NA	
Dieldrin	27.7	29.0	5%	42.7	49.3	9.46	70%	49.2	9.54	68%	2%
4,4'-DDE	65.4	63.6	3%	74.5 D	75.6 D	NS	NA	76.5 D	NS	NA	
2,4'-DDD	38.6 D	38.9 D	1%	60.5 D	57.9 D	NS	NA	58.9 D	NS	NA	
4,4'-DDD	104 D	108 D	4%	157 D	149 D	NS	NA	150 D	NS	NA	
2,4'-DDT	34.5 D	34.5 D	0%	83.5 D	84.7 D	NS	NA	83.9 D	NS	NA	
4,4'-DDT	64.8 D	67.6 D	4%	142 D	151 D	9.46	95%	147 D	9.54	52%	58% #
<b><u>SURROGATE RECOVERIES (%)</u></b>											
PCB103	64.0	64.8		82.5 D	92.0 D			82.8 D			
PCB198	73.3	75.9		79.6 D	86.8 D			74.7 D			
<b><u>AROCLORS</u></b>											
1242	14.2 U	14.2 U		13.5 U	13.6 U			13.7 U			
1248	14.2 U	14.2 U		13.5 U	13.6 U			13.7 U			
1254	187	179	4%	169	243	94.7	78%	249	95.4	84%	7%
1260	14.2 U	14.2 U		13.5 U	13.6 U			13.7 U			

U Not detected at or above DL shown  
 D Diluted 10x

## QA/QC SUMMARY

**PROJECT:** Heckathorn Biomonitoring Year 3  
**PARAMETER:** Pesticides  
**LABORATORY:** Battelle/Marine Sciences Laboratory, Sequim, Washington  
**MATRIX:** Water  
**SAMPLE CUSTODY:** Five water samples (multiple containers of each) were received on 2/17/00. All containers were received in good condition. Cooler temperature upon arrival was 0.3°C. Samples were assigned a Battelle Central File (CF) identification number (1466) and were entered into Battelle's log-in system.

### QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limits</u>	
					<u>Target (ng/L)</u>	<u>Achieved (ng/L)</u>
2,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.01
Dieldrin	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.12
4,4'-DDE	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.03
2,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.03
4,4'-DDD	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.05
2,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.05
4,4'-DDT	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	5	0.05
PCB Aroclor 1242	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	50	14.2
PCB Aroclor 1248	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	50	14.2
PCB Aroclor 1254	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	50	14.2
PCB Aroclor 1260	MeCl <sub>2</sub>	GC-ECD	40-120%	±30%	50	14.2

**METHOD:** Water samples for analysis of chlorinated pesticides and PCBs were processed according to Battelle SOP MSL-O-010, *Extraction and Clean-Up of Water for Surrogate Internal Standard Method*. Water samples were extracted with methylene chloride. Interferences were removed by aluminum/silicon column chromatography. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column (DB-1701) gas chromatography with electron-capture detection (GC/ECD) according to SOP MSL-O-004, *Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection*, which is based on EPA Method 8081 (EPA 1986).

**HOLDING TIMES:** All pesticide extractions and analyses were conducted within target holding times: 14 days to extraction, and 40 days to analysis after extraction. Samples were received on 2/17/00 and held at 4°C. Samples were extracted on 2/18/00 through 2/22/00 and analyzed on 3/21/00. (Water samples were processed immediately to meet holding time requirements, but were held for analysis until corresponding tissue samples were ready for analysis).

**DETECTION LIMITS:** Detection limits for organics were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.

Sample detection limits are calculated using the achieved detection limit and the sample volume.

## QA/QC SUMMARY

### BLANKS/BLANK SPIKES:

One procedural blank and two blank spikes were analyzed. Three analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were spiked into the samples at concentrations of 13.2 ng/L dieldrin and 4,4'-DDT in blank spike A and 13.7 ng/L dieldrin and 4,4'-DDT in blank spike B. Aroclor 1254 was spiked into the blank spikes A and B at 132 ng/L and 137 ng/L, respectively.

All analytes were undetected except 4,4'-DDE and 2,4'-DDT in the dissolved blank. Samples with 4,4'-DDE and 4,4'-DDT detected at concentrations less than 5 times their blank values (0.5 ng/L and 0.65 ng/L, respectively) were flagged with a "B".

Blank spike recoveries were within of the target range of 40%-120% for dieldrin and Aroclor 1254 in both blank spikes A and B. Recovery of 4,4'-DDT was slightly outside the recovery limits in total blank spike A (143%) and within recovery limits in blank spike B.

Precision of the blank spikes replicate analysis, expressed as the RPD between the two replicates, was within the QC limits of  $\pm 30\%$  for dieldrin, 4,4'-DDT, and Aroclor 1254.

### MATRIX SPIKES AND MATRIX SPIKE DUPLICATES:

A matrix spike and matrix spike duplicate (MS/MSD) were prepared and analyzed using two additional samples of sample 303.3. Three analytes of interest, dieldrin, 4,4'-DDT, and Aroclor 1254, were spiked into the total samples at concentrations of 12.3 ng/L dieldrin and 4,4'-DDT in the MS and 12.7 ng/L dieldrin and 4,4'-DDT in the MSD. Aroclor 1254 was spiked into the samples at 123 ng/L in the MS and 133 ng/L in the MSD. Recoveries of dieldrin and 4,4'-DDT could not be calculated because the spike concentration selected was too low relative to the native concentrations of dieldrin and 4,4'-DDT in the sample. Recoveries of Aroclor 1254 could be calculated in the MS but were outside QC criteria. The poor recovery results can likely be attributed to the high and extremely inhomogeneous native levels of dieldrin, 4,4'-DDT, and Aroclor 1254 in the sample. Concentrations of dieldrin and 4,4'-DDT were 50-100 times higher in the sample than the spike level chosen for these analytes; therefore, calculation of recovery was not feasible.

### REPLICATES:

Three field replicate samples were provided for four samples. Relative standard deviation (RSD) between the three field replicates is reported in the data summary table. This information is not used to assess precision. However, it should be noted that Sample 303.3 replicate b had concentrations 5 to 100 times greater than in the other two replicates. Replicates a and c replicated acceptably for most compounds. Greater variability is to be expected between field replicates, which are separately collected samples; the presence of suspended sediment in the water could have contributed to the extreme variability.

### SURROGATE RECOVERIES:

Chlorinated compounds PCBs 103 and 198 were added to each sample during the preparation step as surrogates to assess the efficiency of the extraction procedure. Surrogate recoveries were within the target range of 40%-120% with the exception of surrogate PCB 103 in sample 1466-2c (303.2) at 36.2%.

## QA/QC SUMMARY

### REFERENCES:

U.S. EPA. 1986 (Revised 1990). *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters

Samples Received 2/17/00

MSL Code	1466-1a	1466-1b	1466-1c	1466-2a	1466-2b	1466-2c	1466-3a	1466-3b	1466-3c
STATION NO	303.1	303.1	303.1	303.2	303.2	303.2	303.3	303.3	303.3
LOCATION									
Matrix	Water	Water	Water	Water	Water	Water	Water	Water	Water
Extraction Date	2/18/00	2/18/00	2/18/00	2/18/00	2/18/00	2/18/00	2/22/00	2/22/00	2/22/00
Dilution								100X	
Analytical Batch	1	1	1	1	1	1	1	1	1
Unit	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
2,4'-DDE	0.37	0.24	0.01 U	0.17	0.15	0.31	0.34	13.1	0.30
Dieldrin	1.65	1.61	1.45	10.5	8.60	7.79	100	1710	66.0
4,4'-DDE	0.36 B	0.24 B	0.41 B	1.23	1.49	1.59	0.01 U	124	2.76
2,4'-DDD	0.38	0.27	0.02 U	4.66	4.00	4.16	15.5	223	14.8
4,4'-DDD	1.03	1.04	0.77	17.2	16.0	14.1	41.1	680	45.7
2,4'-DDT	0.20	0.26	0.29	0.92	1.40	1.56	8.28	872	4.49
4,4'-DDT	0.64 B	0.72	0.43 B	3.86	5.85	5.17	17.5	3240	16.7
<b><u>SURROGATE</u></b>									
<b><u>RECOVERIES (%)</u></b>									
PCB103	45.9	53.1	51.0	62.4	67.2	36.2	67.6	83.0	58.9
PCB198	41.9	58.8	46.6	73.8	86.0	41.2	98.3	105	77.2
<b><u>AROCLORS</u></b>									
1242	13.3 U	14.6 U	12.7 U	13.5 U	14.0 U	14.0 U	13.3 U	40.6 U	14.0 U
1248	13.3 U	14.6 U	12.7 U	13.5 U	14.0 U	14.0 U	13.3 U	40.6 U	14.0 U
1254	13.3 U	14.6 U	12.7 U	18.0	21.7	25.6	45.5	449	29.8
1260	13.3 U	14.6 U	12.7 U	13.5 U	14.0 U	14.0 U	13.3 U	40.6 U	14.0 U

U Not detected at or above DL shown

B Concentration is less than 5x blank value

D Diluted 10x

**BATTELLE MARINE SCIENCE**  
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 Sequim, WA 98382-9099  
 360/681-3687

**UNITED HECKATHORN**  
 Pesticides and PCBs in Waters  
 Samples Received 2/17/00

	spk a	spk b				
MSL Code	1466-3d	1466-3e	1466-4a	1466-4b	1466-4c	1466-5
STATION NO	303.3	303.3	303.4	303.4	303.4	303.5
LOCATION						
Matrix	Water	Water	Water	Water	Water	Water
Extraction Date	2/22/00	2/22/00	2/22/00	2/22/00	2/22/00	2/22/00
Dilution	10X					
Analytical Batch	1	1	1	1	1	1
Unit	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
2,4'-DDE	0.01 U	0.44	0.07	0.01 U	0.09	0.04
Dieldrin	229	122	2.68	2.16	1.50	0.91
4,4'-DDE	6.13	2.44	0.46 B	0.39 B	0.39 B	0.25 B
2,4'-DDD	24.6	17.5	0.64	0.58	0.40	0.23
4,4'-DDD	0.04 U	50.0	1.99	1.39	1.03	0.74
2,4'-DDT	0.04 U	2.78	0.37	0.29	0.23	0.17
4,4'-DDT	99.5	25.3	1.38	0.82	0.56	0.55
<b><u>SURROGATE</u></b>						
<b><u>RECOVERIES (%)</u></b>						
PCB103	66.1	58.3	64.4	44.2	53.3	66.3
PCB198	71.0	66.3	69.9	48.4	58.2	67.9
<b><u>AROCLORS</u></b>						
1242	13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	13.0 U
1248	13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	13.0 U
1254	170	133	13.6 U	14.1 U	12.7 U	13.0 U
1260	13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	13.0 U

U Not detected at or above  
 B Concentration is less than  
 D Diluted 10x

B.11

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road  
Sequim, WA 98382-9099  
360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters  
Samples Received 2/17/00

MSL Code STATION NO LOCATION Matrix Extraction Date Dilution Analytical Batch Unit	BSA				BSB			
	Blank	Blank	Spike	Percent	Blank	Spike	Percent	
		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
	Water	Water			Water			
	1	1			1			
	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	%
2,4'-DDE	0.01 U	0.13	NS	NA	0.10	NS	NA	
Dieldrin	0.12 U	14.0	13.2	106%	15.2	13.7	111%	5%
4,4'-DDE	0.10	0.01 U	NS	NA	0.85	NS	NA	
2,4'-DDD	0.03 U	0.03	NS	NA	0.03 U	NS	NA	
4,4'-DDD	0.05 U	0.32	NS	NA	0.40	NS	NA	
2,4'-DDT	0.05 U	0.35	NS	NA	0.38	NS	NA	
4,4'-DDT	0.13	19.0	13.2	143%	# 14.8	13.7	107%	29%
<b><u>SURROGATE RECOVERIES (%)</u></b>								
PCB103	60.6	64.4			63.6			
PCB198	70.7	69.7			61.9			
<b><u>AROCLORS</u></b>								
1242	14.2 U	14.0 U	NS	NA	14.6 U	NS	NA	
1248	14.2 U	14.0 U	NS	NA	14.6 U	NS	NA	
1254	14.2 U	141	132	107%	146	137	107%	0%
1260	14.2 U	14.0 U	NS	NA	14.6 U	NS	NA	

B.12

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters

Samples Received 2/17/00

B.13

		MSA			MSB			
MSL Code	1466-3 <sup>(b)</sup>	1466-3d	Spike	Percent	1466-3e	Spike	Percent	
STATION NO	303.3	303.3			303.3			
LOCATION		Spike A	Amount	Recovery	Spike B	Amount	Recovery	RPD
Matrix	Water	Water			Water			
Extraction Date	2/22/00	2/22/00			2/22/00			
Dilution		10X						
Analytical Batch	1	1			1			
Unit	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	%
2,4'-DDE	4.58	0.01 U	NS	NA	0.44	NS	NA	
Dieldrin	625	229	12.3	SL	122	12.8	SL	NA
4,4'-DDE	42.3	6.13	NS	NA	2.44	NS	NA	
2,4'-DDD	84.4	24.6	NS	NA	17.5	NS	NA	
4,4'-DDD	256	0.04 U	NS	NA	50.0	NS	NA	
2,4'-DDT	295	0.04 U	NS	NA	2.78	NS	NA	
4,4'-DDT	1091	99.5	12.3	SL	25.3	12.8	SL	NA
<b><u>SURROGATE</u></b>								
<b><u>RECOVERIES (%)</u></b>								
PCB103	67.6	66.1			58.3			
PCB198	98.3	71.0			66.3			
<b><u>AROCLORS</u></b>								
1242	13.3 U	13.0 U	NS	NA	13.6 U	NS	NA	
1248	13.3 U	13.0 U	NS	NA	13.6 U	NS	NA	
1254	175	449	123	223% #	133	128	NA	NA
1260	13.3 U	13.0 U	NS	NA	13.6 U	NS	NA	

U Not detected at or above DL shown

D Diluted 10x

SL Inappropriate spike level; see narrative

# Outside QAQC recovery limits

(b) Mean of three reps used to calculate spike recoveries

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters

Samples Received 2/17/00

MSL Code	1466-1a	1466-1b	1466-1c		1466-2a	1466-2b	1466-2c		1466-5
STATION NO	303.1	303.1	303.1		303.2	303.2	303.2		303.5
LOCATION				<i>RSD</i>				<i>RSD</i>	
Matrix	Water	Water	Water		Water	Water	Water		Water
Extraction Date	2/18/00	2/18/00	2/18/00		2/18/00	2/18/00	2/18/00		2/22/00
Dilution									
Analytical Batch	1	1	1		1	1	1		1
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	%	ng/L
2,4'-DDE	0.37	0.24	0.01 U		0.17	0.15	0.31	42% #	0.04
Dieldrin	1.65	1.61	1.45	7%	10.5	8.60	7.79	16%	0.91
4,4'-DDE	0.36	0.24	0.41	26%	1.23	1.49	1.59	13%	0.25
2,4'-DDD	0.38	0.27	0.02 U		4.66	4.00	4.16	8%	0.23
4,4'-DDD	1.03	1.04	0.77	16%	17.2	16.0	14.1	10%	0.74
2,4'-DDT	0.20	0.26	0.29	18%	0.92	1.40	1.56	26%	0.17
4,4'-DDT	0.64 B	0.72 B	0.43 B	25%	3.86	5.85	5.17	20%	0.55
<b><u>SURROGATE</u></b>									
<b><u>RECOVERIES (%)</u></b>									
PCB103	45.9	53.1	51.0		62.4	67.2	36.2		66.3
PCB198	41.9	58.8	46.6		73.8	86.0	41.2		67.9
<b><u>AROCLORS</u></b>									
1242	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U		13.0 U
1248	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U		13.0 U
1254	13.3 U	14.6 U	12.7 U		18.0	21.7	25.6	17%	13.0 U
1260	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U		13.0 U

U Not detected at or above DL shown  
B Concentration is less than 5x blank value  
D Diluted 10x  
# Outside QAQC recovery limits

**BATTELLE MARINE SCIENCES LABORATORY**  
 1529 West Sequim Bay Road  
 Sequim, WA 98382-9099  
 360/681-3687

**UNITED HECKATHORN**  
 Pesticides and PCBs in Waters  
 Samples Received 2/17/00

	spk a		spk b								
MSL Code	1466-3a	1466-3b	1466-3c	1466-3	1466-3d	1466-3e	1466-4a	1466-4b	1466-4c		
STATION NO	303.3	303.3	303.3	303.3	303.3	303.3	303.4	303.4	303.4		
LOCATION	Mean		RSD						RSD		
Matrix	Water	Water	Water		Water	Water	Water	Water	Water		
Extraction Date	2/22/00	2/22/00	2/22/00		2/22/00	2/22/00	2/22/00	2/22/00	2/22/00		
Dilution		100X			10X						
Analytical Batch	1	1	1		1	1	1	1	1		
Unit	ng/L	ng/L	ng/L	ng/L	%	ng/L	ng/L	ng/L	ng/L	ng/L	
2,4'-DDE	0.34	13.1	0.30	4.58	161% #	0.01 U	0.44	0.07	0.01 U	0.09	
Dieldrin	100	1710	66.0	625	150% #	229	122	2.68	2.16	1.50	28%
4,4'-DDE	0.01 U	124	2.76	42.3	168% #	6.13	2.44	0.46	0.39	0.39	10%
2,4'-DDD	15.5	223	14.8	84.4	142% #	24.6	17.5	0.64	0.58	0.40	23%
4,4'-DDD	41.1	680	45.7	256	144% #	0.04 U	50.0	1.99	1.39	1.03	33% #
2,4'-DDT	8.28	872	4.49	295	169% #	0.04 U	2.78	0.37	0.29	0.23	24%
4,4'-DDT	17.5	3240	16.7	1091	170% #	99.5	25.3	1.38	0.82	0.56	46% #
<b><u>SURROGATE</u></b>											
<b><u>RECOVERIES (%)</u></b>											
PCB103	67.6	83.0	58.9			66.1	58.3	64.4	44.2	53.3	
PCB198	98.3	105	77.2			71.0	66.3	69.9	48.4	58.2	
<b><u>AROCLORS</u></b>											
1242	13.3 U	40.6 U	14.0 U			13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	
1248	13.3 U	40.6 U	14.0 U			13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	
1254	45.5	449	29.8	175	136% #	170	133	13.6 U	14.1 U	12.7 U	
1260	13.3 U	40.6 U	14.0 U			13.0 U	13.6 U	13.6 U	14.1 U	12.7 U	

U Not detected at or above DL shown  
 B Concentration is less than 5x blank value  
 D Diluted 10x  
 # Outside QAQC recovery limits

**BATTELLE MARINE SCIENCES LABORATORY**

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters

Samples Received 2/17/00

MSL Code	1466-1a	1466-1b	1466-1c		1466-2a	1466-2b	1466-2c	
STATION NO	303.1	303.1	303.1		303.2	303.2	303.2	
LOCATION				<i>RSD</i>				<i>RSD</i>
Matrix	Water	Water	Water		Water	Water	Water	
Extraction Date	2/18/00	2/18/00	2/18/00		2/18/00	2/18/00	2/18/00	
Dilution								
Analytical Batch	1	1	1		1	1	1	
Unit	ng/L	ng/L	ng/L		ng/L	ng/L	ng/L	%
2,4'-DDE	0.37	0.24	0.01 U		0.17	0.15	0.31	42% #
Dieldrin	1.65	1.61	1.45	7%	10.5	8.60	7.79	16%
4,4'-DDE	0.36	0.24	0.41	26%	1.23	1.49	1.59	13%
2,4'-DDD	0.38	0.27	0.02 U		4.66	4.00	4.16	8%
4,4'-DDD	1.03	1.04	0.77	16%	17.2	16.0	14.1	10%
2,4'-DDT	0.20	0.26	0.29	18%	0.92	1.40	1.56	26%
4,4'-DDT	0.64 B	0.72 B	0.43 B	25%	3.86	5.85	5.17	20%
<b><u>SURROGATE</u></b>								
<b><u>RECOVERIES (%)</u></b>								
PCB103	45.9	53.1	51.0		62.4	67.2	36.2	
PCB198	41.9	58.8	46.6		73.8	86.0	41.2	
<b><u>AROCLORS</u></b>								
1242	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U	
1248	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U	
1254	13.3 U	14.6 U	12.7 U		18.0	21.7	25.6	17%
1260	13.3 U	14.6 U	12.7 U		13.5 U	14.0 U	14.0 U	

U Not detected at or above DL shown  
 B Concentration is less than 5x blank value  
 D Diluted 10x  
 # Outside QAQC recovery limits

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1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3687

**UNITED HECKATHORN**

Pesticides and PCBs in Waters

Samples Received 2/17/00

B.17

MSL Code	1466-3a	1466-3b	1466-3c	1466-3		1466-4a	1466-4b	1466-4c		1466-5
STATION NO	303.3	303.3	303.3	303.3		303.4	303.4	303.4		303.5
LOCATION				Mean	RSD				RSD	
Matrix	Water	Water	Water			Water	Water	Water		Water
Extraction Date	2/22/00	2/22/00	2/22/00			2/22/00	2/22/00	2/22/00		2/22/00
Dilution		100X								
Analytical Batch	1	1	1			1	1	1		1
Unit	ng/L	ng/L	ng/L	ng/L	%	ng/L	ng/L	ng/L		ng/L
2,4'-DDE	0.34	13.1	0.30	4.58	161% #	0.07	0.01 U	0.09		0.04
Dieldrin	100	1710	66.0	625	150% #	2.68	2.16	1.50	28%	0.91
4,4'-DDE	0.01 U	124	2.76	42.3	168% #	0.46	0.39	0.39	10%	0.25
2,4'-DDD	15.5	223	14.8	84.4	142% #	0.64	0.58	0.40	23%	0.23
4,4'-DDD	41.1	680	45.7	256	144% #	1.99	1.39	1.03	33% #	0.74
2,4'-DDT	8.28	872	4.49	295	169% #	0.37	0.29	0.23	24%	0.17
4,4'-DDT	17.5	3240	16.7	1091	170% #	1.38	0.82	0.56	46% #	0.55
<b><u>SURROGATE</u></b>										
<b><u>RECOVERIES (%)</u></b>										
PCB103	67.6	83.0	58.9			64.4	44.2	53.3		66.3
PCB198	98.3	105	77.2			69.9	48.4	58.2		67.9
<b><u>AROCLORS</u></b>										
1242	13.3 U	40.6 U	14.0 U			13.6 U	14.1 U	12.7 U		13.0 U
1248	13.3 U	40.6 U	14.0 U			13.6 U	14.1 U	12.7 U		13.0 U
1254	45.5	449	29.8	175	136% #	13.6 U	14.1 U	12.7 U		13.0 U
1260	13.3 U	40.6 U	14.0 U			13.6 U	14.1 U	12.7 U		13.0 U

U Not detected at or above DL shown  
 B Concentration is less than 5x blank value  
 D Diluted 10x  
 # Outside QAQC recovery limits

APPENDIX C

MUSSEL SHELL LENGTH

RAW DATA

# Resident Mussels Only

Sample ID Battelle Code	Shell Length (cm to nearest 0.01 cm)			
	303.10 20212-Y3M-01	303.40 20212-Y3M-02	303.20 20212-Y3M-03	303.30 20212-Y3M-04
1	3.63	3.01	3.64	4.37
2	3.93	3.80	3.83	4.60
3	3.96	4.00	3.94	4.61
4	4.05	4.00	4.06	4.72
5	4.05	4.03	4.30	4.77
6	4.12	4.05	4.36	4.84
7	4.12	4.10	4.42	4.87
8	4.17	4.20	4.43	4.88
9	4.21	4.20	4.57	4.93
10	4.30	4.21	4.57	4.96
11	4.33	4.21	4.66	4.97
12	4.34	4.22	4.66	4.97
13	4.40	4.24	4.67	5.10
14	4.41	4.24	4.70	5.21
15	4.42	4.27	4.72	5.21
16	4.42	4.40	4.72	5.24
17	4.44	4.45	4.72	5.27
18	4.44	4.50	4.80	5.38
19	4.50	4.65	4.80	5.40
20	4.56	4.65	4.83	5.60
21	4.61	4.66	4.90	5.70
22	4.65	4.70	5.00	5.79
23	4.72	4.82	5.12	5.83
24	4.80	4.83	5.20	5.84
25	4.81	4.88	5.20	5.93
26	4.97	5.00	5.45	6.18
27	5.05	5.03	5.48	6.22
28	5.10	5.03	5.57	6.30
29	5.14	5.05	5.80	6.31
30	5.27	5.22	5.91	6.33
31	5.31	5.27	5.93	6.38
32	5.32	5.31	6.08	6.52
33	5.40	5.43	6.09	6.52
34	5.50	5.44	6.13	6.55
35	5.56	5.50	6.20	6.58
36	5.70	5.53	6.39	6.59
37	5.72	5.73	6.40	6.64
38	5.75	5.80	6.44	6.73
39	5.78	5.82	6.50	6.76

# Resident Mussels Only

Sample ID Battelle Code	Shell Length (cm to nearest 0.01 cm)			
	303.10 20212-Y3M-01	303.40 20212-Y3M-02	303.20 20212-Y3M-03	303.30 20212-Y3M-04
40	5.84	6.05	6.60	6.77
41	5.88	6.10	6.70	7.07
42	5.95	6.19	6.76	7.66
43	6.10	6.56	6.91	
44	6.11	7.00	6.93	
45	6.22		7.06	
46	6.60			
47				
48				
49				
50				
n	46	44	45	42
min	3.63	3.01	3.64	4.37
max	6.60	7.00	7.06	7.66
ratio min:max				
mean length	4.93	4.87	5.34	5.74
	0.74	0.82	0.95	0.82
wet weight (g)				
jar+sample	464.10	473.81	588.05	777.45
jar	312.15	312.61	311.27	432.06
sample only	151.95	161.20	276.78	345.39
n	46	44	45	42
mean wt/mussel	3.30	3.66	6.15	8.22
mean wt/mean size				
mean weight (total)	5.34			

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